

PATENT SPECIFICATION

DRAWINGS ATTACHED

Inventors: HERMANN MAI and HEINZ WAGNER

L147.667



L147.667

Date of Application and filing Complete Specification: 3 Nov., 1966.

No. 49375/66.

Complete Specification Published: 2 April, 1969.

© Crown Copyright 1969.

Index at acceptance:—H1 D(9L, 9Y, 10, 15B, 21C, 21D, 51)

Int. Cl.:—H 01 j 39/36

COMPLETE SPECIFICATION

Improvements in or relating to Mass Spectrometers

- We, FRIEDRICH - SCHILLER - UNIVERSITÄT, of 1, Goetheallee, 69 Jena, Germany, an Undertaking organised under the laws of Eastern Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
- 5 The invention relates to mass spectrometers of the kind which register mass spectra by employing electron-sensitive layers.
- 10 It is an object of the invention to provide a method and a device with which, by using an image converter electrode, the following can be achieved:
- 15 —improved sensitivity resulting from an increase of beam current densities and beam currents;
- 20 —substantial elimination of the background blackening appearing on the recording instrument in the vicinity of the most intense mass lines when a larger number of lines is being recorded simultaneously in a mass
- 25 spectroscopic analysis;
- improved measuring accuracy of this type of analyzing procedure.
- An image converter electrode has already been proposed for use with mass spectrometers equipped with electrometric recording, in order to increase sensitivity. The said electrode is, however, located outside the analyzer field, and the secondary electrons are not subject to any magnetic guide field.
- 30 The arrangement, moreover, allows the recording of several mass lines in temporal or chronological sequence only.
- The ion conversion effect is likewise employed to increase sensitivity in a previously proposed specially designed mass spectrometer with photographic recording. The combination of ion analyzer and image converter electrode used in the process achieves, however, an increase in sensitivity just by utilizing the secondary emission process and the increased sensitivity to electrons of the photographic plate used for spectra recording.
- In mass spectrometers with photographic recording, a certain section of the background blackening is already influenced in the vicinity of the most intense lines of the mass spectrum by means of an electrostatic suppressor plate inserted between photographic plate and analyzer. This arrangement will actually reduce background blackening near the most intense lines of the mass spectrum to some extent, but the remaining amount of this type of blackening, will still constitute a significant mass spectrum interference. In order to reduce background blackening, suggestions have been made to provide the photographic plate with a conductive layer at intense line locations, but the application of such conductive layers has not resulted in an improvement for all elements.
- Moreover a conductive layer placed between a supporting plate and a photographic emulsion results in only a partial suppression of the blackened spots.
- The well known method of intercepting the most intense ion beams in front of the photographic plate by means of one or more miniature Faraday cages can be performed in special cases only.
- Complete suppression of these blackened spots is obtained by sub-dividing the photographic plate, which is mounted in a modified plate holder allowing the most intense ion beams to penetrate freely to the wall of the magnetic analyzer. Since the photographic plate will have to be sub-divided with a

[Price 4s. 6d.]

glazier's diamond in a completely dark room this, however, is an awkward procedure.

The whole ion current is measured indirectly by means of a slit diaphragm before entering the mass separator, and used as a relative measure for the number of singly charged atomic ions of the parent substance trapped by the photographic plate.

The ion current of the singly ionized atoms of the parent substance is measured electrometrically by a special arrangement, and at the same time a section of the mass spectrum is photographically recorded. The application of the whole ion current as a relative measure for the current of singly charged atomic ions leads to errors in the analytical results, as soon as the fractions of the individual beam components generated by the parent substance change.

The other well-known method of electrometrically measuring the ion current of the singly charged ions of the parent substance after their escape from the magnetic field, with simultaneous exposure of the photographic plate will, however, cover only the mass range of the spectrum that is below the atomic mass of the parent substance.

The present invention is based on the desirability of providing a method and a device suitable for increasing beam current density and beam currents, in order to improve detection limit sensitivity and measuring accuracy with mass spectrometers, and for eliminating the blackened spots occurring in the vicinity of the most intense mass lines.

The invention consists in a method of mass spectrometry performed in a mass spectrometer serving to record mass spectra, wherein a primary ion spectrum falls upon an image converter electrode located within a magnetic analyzer field of the spectrometer, the secondary electrons, which are ejected in the ordinarily known way by primary ions at the image converter electrode, being accelerated by a voltage applied between the image converter electrode and an electron-sensitive layer and producing a distinct scaled-down image of the primary ion spectrum by projecting the secondary electrons on the electron-sensitive layer by means of the magnetic analyzer field.

The image converter electrode is desirably arranged in the beam path of the spectrometer at an angle of less than 45° to the focusing plane, this arrangement resulting in increased electron current density.

One or more secondary electron emitter foils, which further increase the secondary electron current emitted by the image converter electrode, may be placed in front of the electron-sensitive layer. The secondary electron emitter foils separate the analyzer chamber from the chamber housing the electron-sensitive layer, so that a pressure dif-

ference can be kept up between the two chambers within certain limits.

In order to facilitate suppression or elimination of the background blackening occurring in mass spectroscopic analyses in the vicinity of the most intense mass lines, one or more portions of the image converter electrode are removed from the beam path at the locations of the most intense mass lines, so that the most intense ion beams are able to pass through the image converter electrode without striking it.

After passing the image converter electrode plane, the most intense ion beams can be intercepted in one or more Faraday cages or by one or more secondary electron multipliers. This arrangement permits direct measurement of the ion currents of certain mass lines during the exposure of the electron-sensitive layer to the remaining mass spectrum. The employment of secondary electrons for recording the primary ion spectrum allows utilization of the higher measuring accuracy obtainable with electron-sensitive layers in comparison with ion sensitive layers.

The electron-sensitive layer may be efficiently calibrated by a separate electron source, the electron energy of which is adjusted to equal the energy of the accelerated secondary electrons.

It will be appreciated that beam current density is increased by secondary electron emission and as a result of the scaling-down effected by the projection. The employment of secondary electron emitter foils, on the other hand, results in further intensification of the electron current. Added to this is the facility of recording electrons with electron-sensitive layers at a higher level of sensitivity than that applied to recording ions. This feature results in a considerable decrease of the analyzing time compared with the conventional method. At the same time the secondary electron emitter foils allow the analyzer chamber to be separated from the chamber which contains the electron-sensitive layer; this arrangement permits pressure reduction in the analyzer chamber. The pressure reduction in the analyzer chamber results in a lowered spectrum background and consequently in improved detection limits.

Moreover the interaction of the most intense ion beams with the image converter electrode is inhibited during the whole analyzing exposure, and direct measurement of the ion current of certain ion beams is possible during every single exposure. Background blackening intensity of mass spectra decreases in the vicinity of the most intense lines to an extent resulting in improved detection limits for many elements of more than one order of magnitude. The arrangement permits also a significant extension of the exposure interval towards larger exposures,

which in turn results in improved detection limits for all other elements.

Electrometric measurement of one or more defined ion beams with simultaneous exposure of the electron-sensitive layer to the mass spectra by the secondary electrons is possible, and this results in a reduction of measuring errors as compared with conventional methods, namely by direct measurement of the singly charged atomic ions of the parent substance on the one hand and the increased measuring accuracy, which distinguishes the electron-sensitive layers employed from the ion-sensitive layers, on the other.

For recording the secondary electrons beams, electron-sensitive plates or films can be used that are less liable to mechanical damage than the photographic plates of the Schumann type at present in common use for ion recording.

In order to make the invention clearly understood, reference will now be made to the accompanying drawings which are given by way of example and in which:—

Fig. 1 is a diagrammatic perspective view of the image converter electrode arrangement in a mass spectrometer; and

Fig. 2 is a sectional view of the arrangement shown in Fig. 1.

Ion beams 1 emitted by an ion source fall upon an image converter electrode 3, which is mounted between pole pieces 2, provided with a slit 3', and arranged at an angle of less than 45° to the focusing plane 4. The impact of ion beams 1 on the surface of image converter electrode 3 releases secondary electrons. These are accelerated by the applied electric field U_c towards an electron-sensitive layer 5 on a supporting base 6, with the magnetic field of pole pieces 2 providing guidance for the secondary electrons in the direction of the lines of magnetism. Above the image converter electrode 3 there are a slit diaphragm 7 and a secondary electron emitter foil 8, which are penetrated by the secondary electron before they strike the electron-sensitive layer 5. A component of the ion beam 1 representing the most intense mass line of the ion spectrum passes through slit 3' of the image converter electrode 3 and is intercepted for electrometric measurement by a Faraday cage 9.

WHAT WE CLAIM IS:—

1. A method of mass spectrometry performed in a mass spectrometer serving to record mass spectra, wherein a primary ion spectrum falls upon an image converter electrode located within a magnetic analyser field of the spectrometer, the secondary electrons, which are ejected in the ordinarily known way by primary ions at the image converter electrode, being accelerated by a voltage applied between the image converter electrode

and an electron-sensitive layer and producing a distinct scaled-down image of the primary ion spectrum by projecting the secondary electrons on the electron-sensitive layer by means of the magnetic analyser field.

2. A method as claimed in Claim 1, in which the secondary electron current emitted by the image converter electrode is further increased by one or more secondary electron emitter foils arranged in front of the electron-sensitive layer.

3. A method as claimed in Claim 2, in which an analyser chamber is separated from a chamber housing the electron-sensitive layer by the secondary electron emitter foils, and a pressure difference is maintained between the two chambers.

4. A method as claimed in Claim 1, in which one or more portions of the image converter electrode are removed from the beam path at the locations of the most intense mass-lines in order to facilitate suppression or elimination of background blackening.

5. A method as claimed in Claim 4, in which the ion currents of the most intense mass lines, which are allowed to pass the image converter electrode plane at the locations where portions of the converter electrode are removed, are directly measured electrometrically during the exposure of the electron-sensitive layer to the remaining ion spectrum.

6. A method as claimed in Claim 1, in which the electron-sensitive layer is calibrated by a separate electron source.

7. A mass spectrometer for use in performing the method of Claim 1, comprising an image converter electrode disposed within an analyser field of the spectrometer at an angle of less than 45° to the focusing plane of the spectrometer, means being provided for carrying a recording medium in the form of an electron-sensitive layer for being subjected to secondary electrons ejected from the image converter electrode by primary ions, means for applying a voltage between the image converter electrode and the electron-sensitive layer being provided for accelerating the secondary electrons, the arrangement being such that the secondary electrons projected onto the electron-sensitive layer produce a scaled-down image of the primary ion spectrum.

8. A mass spectrometer as claimed in Claim 7, wherein one or more secondary electron emitter foils are arranged in the electron beam in front of the electron-sensitive layer.

9. A mass spectrometer as claimed in Claim 7 or 8, wherein the image converter electrode is provided with one or more slits.

10. A mass spectrometer as claimed in Claim 9, wherein one or more Faraday cages, or one or more secondary electron multipliers, are provided behind the slits of the image converter electrode.

11. A mass spectrometer constructed and arranged substantially as hereinbefore described with reference to and as illustrated in the accompanying drawings.

VENNER, SHIPLEY & CO.,
Chartered Patent Agents.
Rugby Chambers, 2 Rugby Street,
London, W.C.1.
Agents for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1969.
Published by the Patent Office, 25, Southampton Buildings, London, W.C.2, from which copies may be obtained.

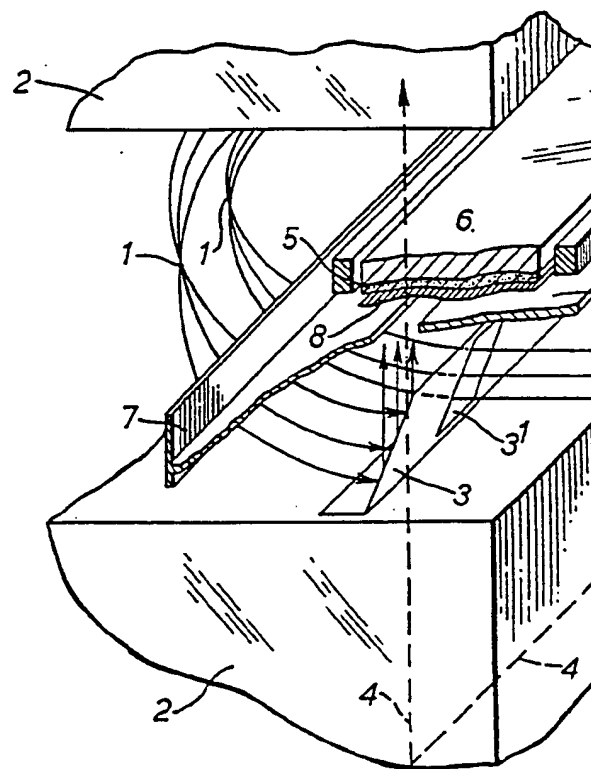


FIG. 2

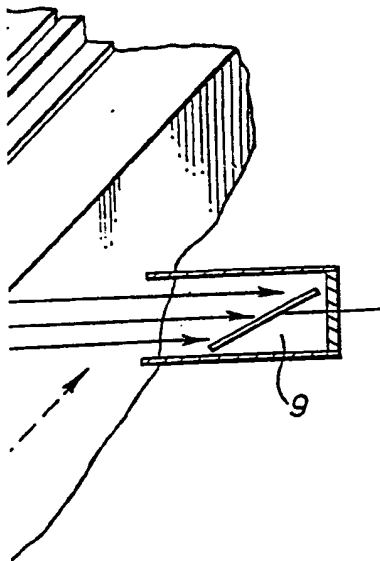
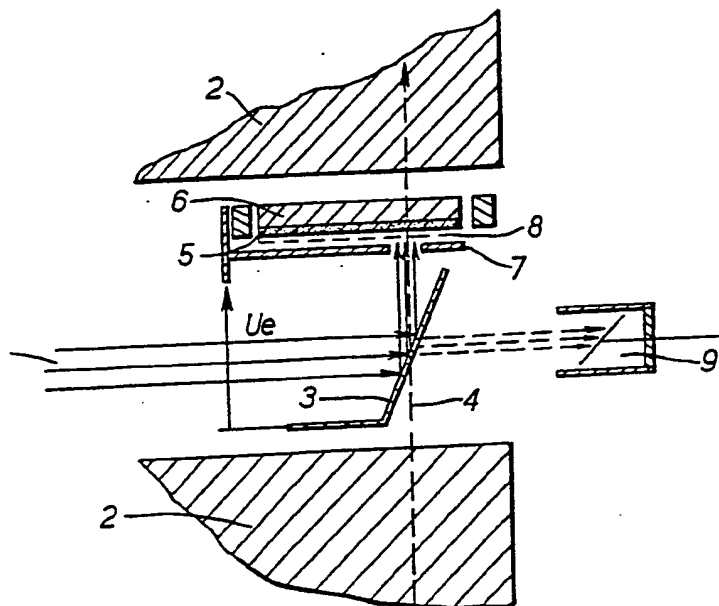


FIG. 1



1147557 COMPLETE SPECIFICATION
This drawing is a reproduction of
the Original on a reduced scale

